Benzotriazole-Assisted Thioacylation

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Crystal data for bis(benzotriazolyl)-bis(phenylthio)methane (4a): C₂₅H₁₈N₆S₂, MW 466.57, orthorhombic, Pbca, $a = 7.322(1)$, $b = 21.448(4)$, $c = 28.651(5)$ Å, $V = 4499(1)$ Å³, $Z = 8$, $T = -110$ °C, $F(000) = 1936$, μ (MoK α) = 0.263 mm⁻¹, $D_{\text{calcd}} = 1.378$ g.cm⁻³, $2\theta_{\text{max}} 50^\circ$ (CCD area detector, 100% completeness), $wR(F^2) = 0.0596$ (all 3963 data), $R = 0.0361$ (2002 data with $I > 2\sigma I$).

Crystal data for bis(benzotriazolyl)-bis(4-methylphenylthio)methane (4b): C₂₇H₂₂N₆S₂, MW 494.64, monoclinic, P2₁/n, $a = 7.828(1)$, $b = 14.795(2)$, $c = 20.827(3)$ Å, $\beta = 96.982(2)^\circ$, $V = 2394.2(5)$ Å³, $Z = 4$, $T = -110$ °C, $F(000) = 1032$, μ (MoK α) = 0.252 mm⁻¹, $D_{\text{calcd}} = 1.372$ g.cm⁻³, $2\theta_{\text{max}} 53^\circ$ (CCD area detector, 98.1% completeness), $wR(F^2) = 0.0810$ (all 4866 data), $R = 0.0346$ (3335 data with $I > 2\sigma I$).

Crystal data for isopropyl di(benzotriazol-1-yl)methyl disulfide (14d): C₁₆H₁₆N₂S₂, MW 356.47, triclinic, P-1, $a = 9.864(2)$, $b = 10.258(2)$, $c = 10.408(2)$ Å, $\alpha = 99.580(3)$, $\beta = 107.887(3)$, $\gamma = 115.446(2)^\circ$, $V = 848.7(2)$ Å³, $Z = 2$, $T = -160$ °C, $F(000) = 372$, μ (MoK α) = 0.324 mm⁻¹, $D_{\text{calcd}} = 1.395$ g.cm⁻³, $2\theta_{\text{max}} 50^\circ$ (CCD area detector, 98.0% completeness), $wR(F^2) = 0.0932$ (all 2945 data), $R = 0.0346$ (2628 data with $I > 2\sigma I$).

C₂₅H₁₆N₆S₂
 Exact Mass: 466.59
 Mol. Wt.: 466.57
 C, 64.36; H, 3.89; N, 18.01

X-ray structure of bis(benzotriazol-1-yl)-di-(phenylthio)methane (4a).

$C_{27}H_{22}N_6S_2$

Exact Mass: 494.13

Mol. Wt.: 494.64

C, 65.56; H, 4.48; N, 16.99; S, 12.97

X-ray structure of bis(benzotriazol-1-yl)-di-(4-methylphenylthio)methane (**4b**).

$C_{16}H_{16}N_6S_2$

Mol. Wt.: 356.47

Required: C, 53.91; H, 4.52; N, 23.58

Found: C, 53.73; H, 4.56; N, 23.25

FIGURE 2. X-ray structure of isopropyl di(benzotriazol-1-yl)methyl disulfide (**14d**).